

Structure Determination of a Novel Redox-active Macrocycle Containing the Ferrocene Unit from Powder Diffraction Data Using Simulated Annealing

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Introduction: Recent attention has focused on host molecules that contain responsive elements as an integral part of a macrocyclic framework. Our group is currently developing novel strategies for the generation of molecular cavities possessing electrochemical response functions (e.g. ferrocene units, quinone derivatives). The aim lies in the selective recognition and binding of anionic species. Starting from 1,1'-diborylated ferrocenes [1,1'-fc(BRR')₂] and bifunctional organic linkers [e.g. 2,5-di(1-pyrazolyl)hydroquinone], the facile formation of boron-nitrogen- and boron-oxygen bonds is exploited for the self assembly of macrocyclic architectures.

Methods and Materials X-ray powder diffraction data were collected for FeB₂O₂C₂₄N₄H₂₂ at 295 K ($\lambda = 1.14991(2)$ Å) in transmission geometry with the sample sealed in a 0.7 mm lithium borate glass capillary (**Figure 1**). Data reduction was performed using the GUF1 program. Indexing with ITO led to a C centered monoclinic unit cell with lattice parameters of $a = 14.930$, $b = 11.465$, $c = 24.554$ [Å], and $\beta = 98.969^\circ$. Extinction rules indicated Cc or $C2/c$ as possible space groups, from which the latter could later be confirmed by Rietveld refinement. The crystal structure was solved using the PowderSolve (MSI) structure solution package (**Figure 2**).

Results: X-ray crystallography provides the most powerful analytical tool to verify, that cyclic molecules have been generated, and to exclude, that open-chain polymeric species have been formed. In the present case, numerous attempts to grow single crystals of the title compound failed. Thus, the structure information required had to be determined from high resolution X-ray powder diffraction data.

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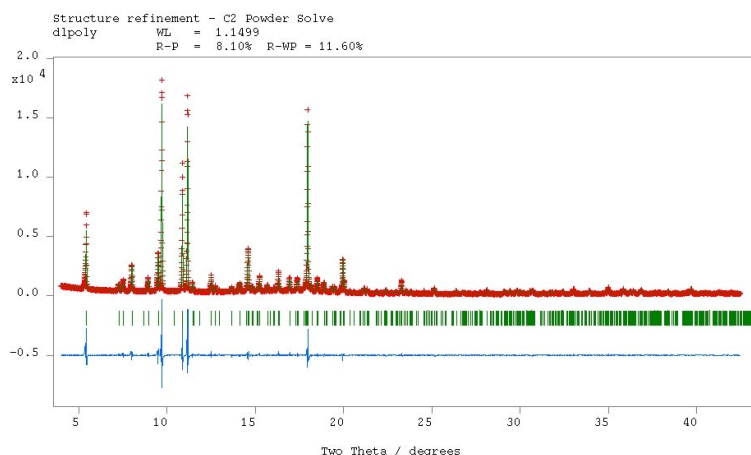


Figure 1. Scattered X-ray intensity for FeB₂O₂C₂₄N₄H₂₂ at ambient conditions as a function of diffraction angle 2θ . Shown are the observed pattern (diamonds), the best Rietveld fit profile (line), the reflection positions and the difference curve between observed and calculated profiles as the trace at the bottom.

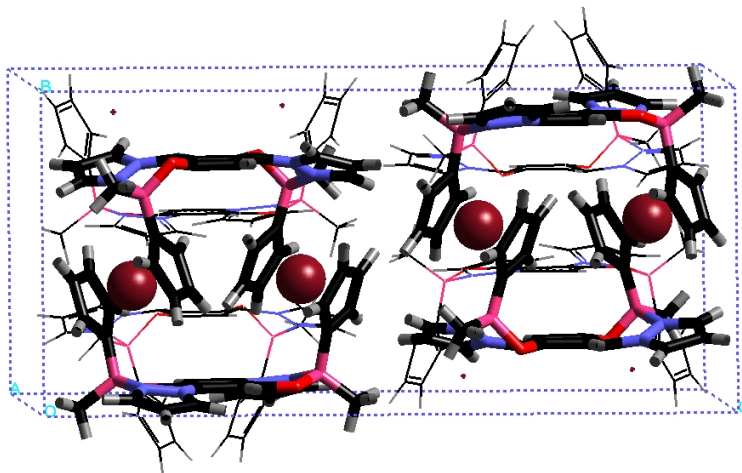


Figure 2. Crystal structure of FeB₂O₂C₂₄N₄H₂₂ in a projection along a -axis